DEHYDRATION OF ALFALFA WITH SUPERHEATED STEAM

by

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INTRODUCTION

The purpose of this research was to compare the effect of dehydrating alfalfa with superheated steam on the carotene content with the effect produced by present drying mediums used for dehydration. Carotene content retention was determined immediately upon completion of dehydration and after storage. Even though a large industry has sprung up during the past few years to supply dehydrated alfalfa, no study of this nature has appeared in literature.

In common driers, air or hot fuel gases are used as the drying medium. Latent heat of vaporization is supplied to the wet material by convection and radiation from heated air or gases. Usual drying mediums such as air have limitations in that they may only be used when no undesirable chemical changes or destruction take place in the material being dried; therefore, other drying mediums may be used to improve these undesirable conditions.

In any drying process a transfer of heat and mass is involved between the drying medium and the wet solid. For many porous solids exposed to constant drying conditions, this process is characterized by two distinct drying rate periods: one during which the rate of drying is constant and one during which the rate is continually decreased. The constant rate period exists at moisture contents sufficient to keep the surface of the solid wet. When the transfer of water within the

solid is insufficient to keep the surface of the solid wet, the
rate of drying decreases as the moisture content decreases. The
use of superheated steam instead of air as a drying medium does
not alter this general characteristic of the drying process (9).

Superheated steam has been shown to be an attractive drying
agent for materials which are not temperature sensitive. Water
removed from the solid during the drying process becomes, on
heating, a part of the drying medium whereas in air drying, sat-
urated air must ultimately be replaced by fresh air heated to a
suitable operating temperature level (9). Thus, use of super-
heated steam for drying favors higher drying rates and thermal
efficiencies than are possible with air. Therefore, use of
steam was justified by savings in operating cost.

Alfalfa, when dehydrated by field curing, loses 50 to 75
percent of its carotene content in harvesting alone, while 5 to
15 percent of the original carotene is lost in a well controlled
dryer using hot fuel gases without undue exposure to the bleach-
ing action of the sun (8). Thus, high carotene content preser-
vation in alfalfa is largely improved by rapid reduction of
moisture content. However, 100 percent preservation has not been
accomplished by the commercial dehydrators.

Griffith and Thompson (1) state that in drying alfalfa,
oxygen and enzyme activity were necessary for carotene destruc-
tion. Mitchell and Houge (3) state that the carotene-destroying
system in alfalfa was enzyme in nature, and if the alfalfa was
frozen and before it was allowed to wilt, enzymic destruction

of carotene was stopped. Mitchell of the chemistry department of Kansas State College[4(a)] stated that if the enzymes were first destroyed in alfalfa, drying of the material could be carried out without carotene destruction. Mitchell also stated that the destruction of the enzymes could be carried out by blanching with steam or water at near 80° centigrade for about five minutes. This blanching did not affect the green coloring of the alfalfa. Hart and Mills (2) stated that upon autoclaving at 15 psia for one hour the carotene content was stable, but that chlorophyll and oxidase was destroyed, leaving a material gray in color.

Griffith and Thompson (1) stated that blanching for long periods (more than five minutes at five pounds psia) destroyed natural antioxidants and decreased stability of carotene in storage. Nelson, Loosli, Lofgreen, and Yager (5) stated that blanching did not affect the retention of carotene during storage; and that when the meal was stored for a period of six months at 40° F. in cloth or paper bags, it showed a rise in water content and a slight rise in carotene content.

This research was formulated and carried out from the foregoing statements. The data here presented and correlated were obtained through the project sponsored by the Kansas Industrial Development Commission on dehydration at Kansas State College.

[4(a)]Mitchell, H. L. Private Communication.

EQUIPMENT

Materials of Construction and Size

The dehydrator, as shown in Plate I, was designed for use on the Kansas Industrial Development Commission's dehydration project at Kansas State College. The drying oven of the dehydrator was a cylinder two feet high by 13-3/4 inches outside diameter constructed of 24 gage sheet aluminum. A 16 inch outside diameter, 20 gage galvanized sheet cylinder, two feet high was placed around the drying oven for insulation. The top of the oven was a 14-1/4 inch outside diameter aluminum lid, 24 gage thick. Suspended from the lid by means of three straps was a 12-1/2 inch outside diameter cylinder, six inches long, which contained a static drying bed and a temperature recording grid. The overall length of the straps and suspended cylinder was one foot. The lid and attached bed support and grid was removable. The bottom of the drying oven was two by two feet and constructed of number 16 gage galvanized iron. The upright section of the oven was not connected to the bottom section by any mechanical device and was constructed so as to set snugly upon the bottom section. At the center of the bottom section was connected a three inch inside diameter duct, constructed of 22 gage galvanized iron, for the entrance of the saturated steam to the drying oven. Immediately above the steam opening was placed a series of baffle plates to distribute the saturated steam uniformly to the upright section of the oven. The baffle

EXPLANATION OF PLATE I

Equipment Used in Dehydrating Alfalfa

1. Powerstat
2. Pressure Gage
3. Throttling Calorimeter
4. Manometer
5. Brown Electronic Recorder
6. Drying Oven
PT-7 Portable Potentiometer
PT-10 General Electrical Self-Balancing Potentiometer
D. Decade Boxes

6

PLATE I

plates were connected to the bottom section and were made of
22 gage galvanized iron, doughnut in shape with a decrease in
diameter as they proceeded in height. A total of six baffle
plates were used with the top baffle having no opening in the
center and being 3/4 inches in diameter. Each baffle plate was
constructed to support the baffle plate placed above by means
of three 1/8 inch phillister head screw bolts and nuts.

Inside of the oven was a spiral of galvanized wire two
feet high by 13-1/2 inches inside diameter. Each spiral was
two inches high, making a total of 12 spirals in the two feet
of length. The spiral of wire was made rigid by brazing three
upright wire post to each spiral placed 120° apart. The wire
spiral extended the length of the upright drying section of the
oven. Inside the wire spiral, as shown in Plate II, was a heat-
ing element. Seven inches above the heating element was placed
a reflection shield which was beneath a temperature recording
grid by a distance of 2-1/2 inches. The heating element, re-
flection shield, and temperature recording grid were held in
place by short pieces of rigid wire brazed to the three upright
wire post of the spiral.

The drying oven was mounted on a stand 18 inches high which
was constructed of angle iron.

The three inch inside diameter duct that was fastened to
the bottom section of the drying oven was constructed in an
L-shape. The section of the ell fastened to the oven was eight
inches long and the other portion of the ell was 14 inches long.
A drain cock was placed at the lowest point of the L-section of

EXPLANATION OF PLATE II

A. Wire spiral

B. Wire post

C. Baffle plate

D. Heater

E. Reflection grid

F. Temperature recording grid

PLATE II
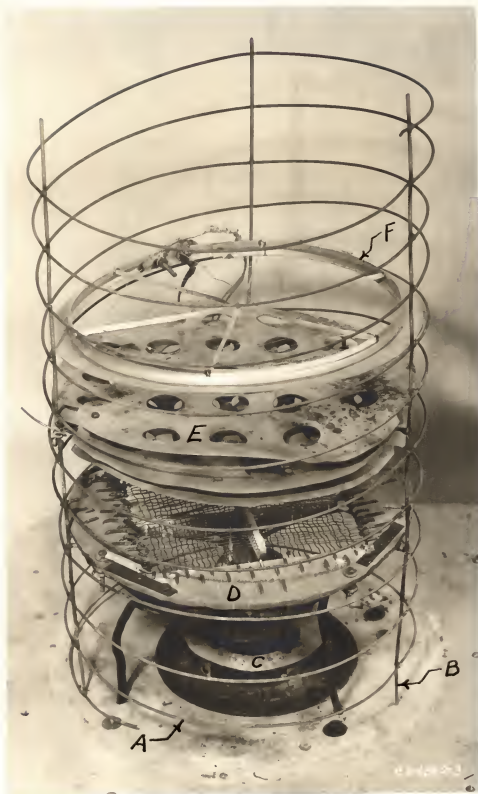
the duct to drain the condensed saturated steam vapors that forms during the initial period of operation.

A one-inch pipe line was connected to the 14 inch section of the L-shaped duct. This pipe line was connected to the high pressure steam line that serves as a source of saturated steam for the experimental laboratory of the department. The one-inch pipe line, as shown in Plate III, contained a "knockout pot" to remove any excess water that was in steam line, a 1/2 inch Mueller pressure regulating valve to obtain a constant steam pressure for feed to oven, a pressure gage and throttling steam calorimeter which was connected to the one-inch pipe line by proper pipe fittings, and a previously calibrated steam orifice plate surmounted by a manometer. The throttling steam calorimeter was used to determine the amount of moisture in the steam, and the steam orifice and manometer was used to determine the rate of flow of the saturated steam. An air line was also connected to the one-inch pipe line immediately following the Mueller pressure valve. Air was used for a drying medium in experimental runs, 26 through 35 to provide a comparison between the two drying mediums.

Equipment used to measure the temperature of the temperature recording grids was a Brown, 16 point, electronic recording potentiometer, two decade resistance boxes, a General Electric self-balancing potentiometer, a portable potentiometer, a double di-pole double throw switch, a standard resistor, and a 1.5 volt dry cell battery.

EXPLANATION OF PLATE III

Flow Diagram

A. Saturated steam from steam supply line

B. "Knockout" Pot

C. Mueller pressure regulater valve

D. Throttling calorimeter

E. Manometer

F. Orifice plate

G. Baffle plates

H. Heater

I. Reflection shield

J. Lower temperature recording grid

K. Static bed

L. Upper temperature recording grid

M. Steam outlet

N. Compressed air inlet

O. Main body of oven

PLATE III

12
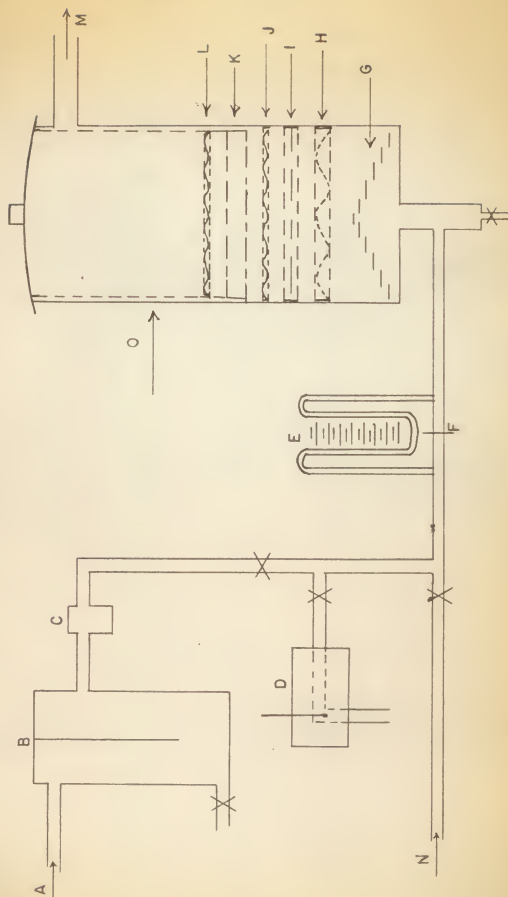
The heating element of the oven was connected to a Superior Electric Company Powerstat by lead wires which were capable of delivering a maximum output of 28 amps at 270 volts from a 220 volt circuit supply. By use of the Powerstat, the amount of voltage to the heating element was controlled.

Auxiliary equipment used included a Waring Blender, Beckman Spectrophotometer, ordinary chemical laboratory equipment for analytical work, vacuum oven, and vacuum pump. The Waring Blender was a standard commercial model and the Beckman Quartz Spectrophotometer was manufactured by National Technical Laboratories, South Pasadena, California. Carotene concentration was measured at 4360 $\mathring{R}$. The vacuum oven was a Weber electric vacuum oven, sold by Arthur H. Thomas Company of Philadelphia, Pennsylvania. The vacuum pump was one-half H.P. made by G. F. Nelson of Berkeley, California. This equipment was used to determine the carotene and moisture content of the alfalfa before and after dehydration.

The chemicals used for the carotent analysis were Skellysolv-B, ethyl alcohol, acetone, an hydrous sodium sulfate, and magnesium oxide.

Design Calculations and Instrumentation

Throttling Steam Calorimeter. If by throttling to atmospheric pressure, wet steam still can be superheated, the amount of moisture in the steam can be determined by the throttling calorimeter. In the throttling process, enthalpy is converted into kinetic energy, which is completely reconverted by friction

into heat without doing external work. Therefore, the enthalpy, before and after throttling, has the same value. Accordingly, the following must be observed:

1. The kinetic energy created by the throttling process must be destroyed and reconverted into heat so that the initial value of enthalpy is restored.

2. The superheated steam must be completely dry. Due to slow heat conduction to droplets suspended in the steam, superheated steam frequently contains moisture. Complete evaporation of the droplets can be obtained by deflecting the steam jet towards a wall.

3. The temperature must be measured at a spot where conditions (1) and (2) are fulfilled.
By meeting the above requirements, the throttling steam calorimeter was designed, Fig. 1.

The steam quality was obtained directly from the Mollier chart by tracing a line of constant enthalpy from the calorimeter temperature and atmospheric pressure to the pressure reading of the pressure gage placed in the steam line before the calorimeter.

One-hundred percent steam quality was obtained after letting steam flow for a period of time. The throttling calorimeter served to locate the time at which experimental drying determinations could be started.

Temperature Recording Grids. The temperature recording grids were constructed of 24 gage aluminum rings, 13 inches in-

THROTTLING
STEAM CALORIMETER

HIGH PRESSURE VALVE ½"

½ PIPE THREAD

INLET

MACHINED ORIFICE NIPPLE

3"

STD COUPLING

½" x 1"

1" x 4" STD. NIPPLE

1" x 1½" STD BUSHING

1½ STD. TEE

RUBBER STOPPER

CALIBRATED THERMOMETER RANGE 200-400° F.

INSULATION

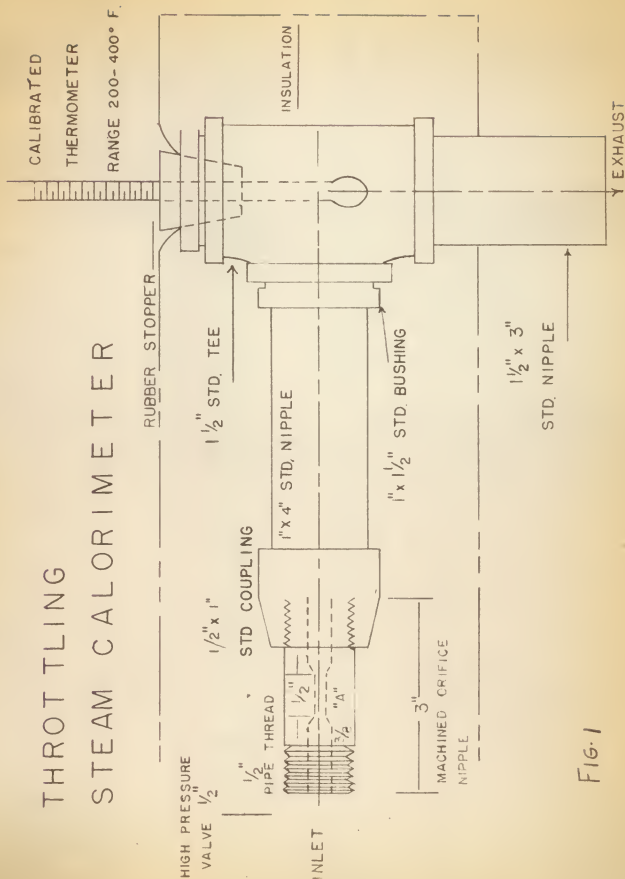EXHAUST

1½" x 3" STD. NIPPLE

FIG. 1

side diameter by 13-1/2 inches outside diameter. Inside of the rings were placed approximately eight feet of silver wire, which was dispersed throughout the diameter of the aluminum rings in the form of a flat spiral. The silver wire was insulated from the aluminum rings by six cords of glass fiber tape extending across the diameter of the aluminum rings. To the ends of the silver wire were attached the lead wires which were connected to the Brown 16-point recorder. The size of the silver wire was approximately 1/128 inch diameter (Plate IV).

Since the standard resistor for use at room temperature was 1.142 ohms resistance, the resistance of the silver wire of each grid was made to have the same approximate resistance at 212° F. This was accomplished by using a piece of silver wire of sufficient length to give the required resistance, the resistance being measured by a Leeds-Northrop Wheatstone Bridge instrument which could be read with accuracy to plus or minus 0.001 ohms.

The temperature resistance coefficient for the silver wire was calculated and found to be different for each grid. This calculation was made as follows:

1. By use of Leeds-Northrop Wheatstone Bridge instrument, the resistance of the wires was measured at 32° F. and 212° F.

2. Resistance of grid above static bed was 0.798 ohms at 32° F. and 1.141 ohms at 212° F.

3. Resistance of grid below static bed was 0.810 ohms at 32° F. and 1.142 at 212° F.

EXPLANATION OF PLATE IV

A. Temperature recording grid

B. Static bed that contained alfalfa

PLATE IV

B

A

0.04862-2
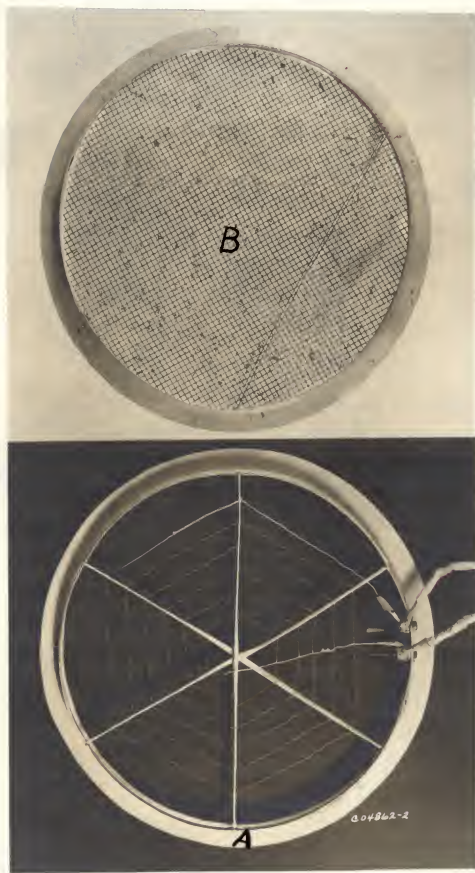
4. By use of equation, $R_t = R_o(1 + \alpha T)$, the α values were
obtained.   Where R_t = Resistance at 212° F.
                     R_o = Resistance at 32° F.
                     T = 212° - 32°

5. α value of top grid was found to be 0.002150 $(°F)^{-1}$,
and of the bottom grid, 0.002315 $(°F.)^{-1}$.

Static Bed. The size of the static bed was determined by
the amount of dehydrated material necessary for the carotene
and moisture content analysis. Four grams each were necessary
for carotene and moisture analysis. An additional 7 or 8 grams
were also considered for an additional analysis in the event of
a mistake in the initial analysis. Since the initial moisture
content of the green alfalfa was approximately 70 percent, an
initial charge of 50 grams were needed to insure the proper de-
hydrated amount of 15 or 16 grams for analysis. Because the
green alfalfa to be dehydrated was to be placed on the bed as a
single layer, the area of the bed needed was approximately 110
square inches. Thus, a bed of 12 inches diameter was con-
structed (Plate IV).

Heating Element. The degree of superheat of the drying
medium was desired to cover a span of 212° F. to 900° F. To
provide this range of superheat, 50.8 feet of Nicrome No. 20
Cromel A resistance wire was needed. This length was calcula-
ted according to the specifications published by the Driver-
Harris Company (6).

The calculations were as follows:

Mass velocity through heating element $= 76 \frac{\text{lb. steam}}{\text{hr.- ft.}}$

Area of static bed = approximately 50 ft.
Assumed   steam   rate = 76 lb. / hr.

Superheat temperature of steam assumed = 900 - 212 = 688° F.

C_{Pm} (900° F) = 8.5 B.T.U./lb mole - ° F.

C_{Pm} (212° F) = 8.05 B.T.U. /lb mole - ° F.

$\frac{Q}{t} = \frac{76}{18} \{ (8.5)(900) - 8.05 (212) \}$

= 76 (7650 - 1710) = 25,000 B.T.U./hr.

Where: C_{Pm} = mean heat capacity of steam
       Q = heat required in B.T.U.
       t = time (hrs.)

1 watt = 14.34 cal/min

1 kilowatt - 14,340 cal/min = $\frac{14,340}{252}$ = 57.0 B.T.U./min

57 B.T.U./min = 3,420 B.T.U./hr.

Then power requirement = $\frac{25,000}{3,420}$ = 7.3 kilowatts

No. of Amps. = $\frac{7,300 \text{ watts}}{270 \text{ volts}}$ = 27 amps.

Since powerstat output rating was 7.5 k.v.a. at 270 volts
or amps the length of wire required for number 20 nichrome
straight wire is as follows:

Calculation for temperature of wire if stretched in par-
allel (two sections).

Current allowed = $\frac{28}{2}$ = 14 amps.

Temperature = 1600 ≠ $\frac{14-12.5}{14.6 - 12.5}$ (1800 - 1600) = 1743° F.

Calculation for length of each wire:

Resistance at 1743° F.

The resistance at 68° F. = 0.6592 ohms/ft.

Therefore the resistance at 1743° F. = 0.6592 (c).

$$= 0.6592 (1.15)$$

$$= 0.758 \text{ ohms/ft.}$$

Where "c" - factor by which resistance at room tempera-
ture is to be multiplied to obtain resist-
ance at indicated temperature.

From ohm's law -

$E = IR$

$270 = 14 R$

$R = \frac{270}{14}$ or 19.28 ohms.

Number of feet of wire required for one section of heat-
ing element $= \frac{19.28}{0.758}$

$$= 25.4 \text{ ft. of wire}$$

Therefore, the total length of wire for bottom and top
sections of heating element = 2 x 25.4 or 50.8 ft.

To insure the insulation of the resistance wire from the
main body of the dehydrator, 1/4 inch thick transite was used.
The outside diameter of the Transite ring was 13-1/2 inches,
and the dimensions used for the 50.8 feet of resistance wire
was 8 inches by 8 inches. This 8 inch square was cut from the
middle of the 13-1/2 inch diameter piece of transite. The 50.8
feet of resistance wire was held in place by wire hooks embedded

in the transite on each of the sides of the eight inch square.
This produced the two grids of wire, one grid below the other
with the wire of each grid running perpendicular in direction
to the wire of the other grid. The wire of each grid was ap-
proximately 1/8 inch apart and was held in this position by
a spacer. The spacer for each grid was held in place by springs
which also served to take up any slack that took place when the
resistance wire was heated (Plate V). Necessary post for con-
nection were constructed of 1/8 inch copper bolts.

When the saturated steam was passed through the heating
element, the steam was given the desired degree of superheat.
The degree of superheat was controlled by the amount of cur-
rent passed through the resistance heater which, in turn, was
regulated by the Powerstat.

Since the design of the heating element was based upon an
assumed flow of 76 lb./hr. of steam, a device to control the
rate of steam flow was inserted in the stem line. This device
was an orifice plate, surmounted by a manometer to measure the
difference in pressure between the upstream and downstream
section of the orifice plate.

The orifice plate used was originally designed and cali-
brated for use in air flow; therefore, the orifice coefficient
for steam flow was unknown. This orifice coefficient was cal-
culated for steam flow from the actual results obtained by
using the orifice under the same steam flow conditions that was
applied to the experimental study.

PLATE V
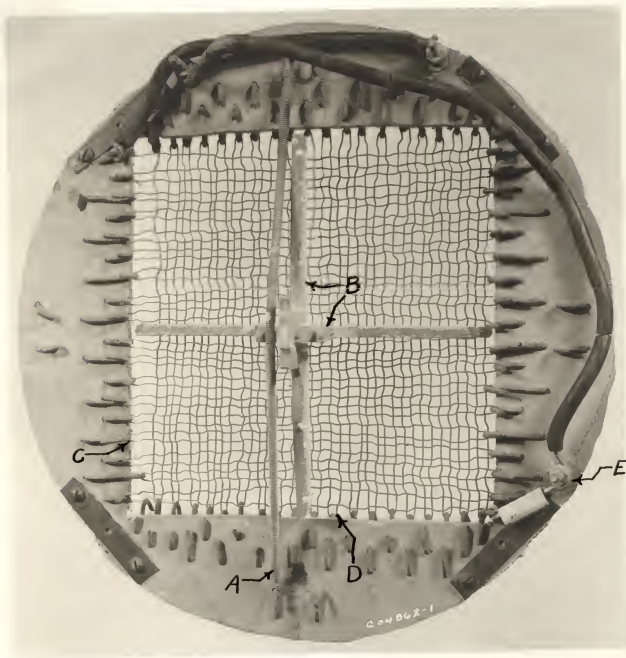
By the use of a condensor, the maximum rate of flow of steam in pounds per hour was measured at different line pressures. With the maximum rate of flow known, the orifice coefficient was calculated. The following equation was used:

$$C = \frac{Q}{AD^2 HKK_1}$$

Where C = orifice coefficient

Q = maximum rate of flow of steam in pounds per hour

A = numerical factor dependent on unit of flow used and in this case is 1272

D = diameter of orifice, in this -.533 inches

H = square root of maximum pressure differential, inches of mercury under water.

K = density factor, square root of dry saturated steam or square root of pound per cubic foot.

K_1 = quality of steam which was 100%

For the values of "Q" determined, the orifice coefficient for steam was calculated to equal 0.1400. With the orifice coefficient for steam flow known, the rate of steam flow in pounds per hour was calculated at different pressure differentials until the desired rate of 80 pounds per hour was known. At this flow rate the line pressure was 2.5 pounds per square inch and the pressure differential across the manometer was 4.31 inches of mercury under water.

Brown 16-point Electronic Recording Potentiometer. The temperatures measured by the temperature recording grids were

recorded by a Brown, 16-point, Electronic Recording Potentio-
meter. Initially this instrument was constructed to measure
temperatures by means of thermo couples. To better suit the
needs of this research the instrument was converted to read
0-100 millivolts by alternately measuring the potential drop
across an external standard resistance, the lower bed tempera-
ture grid and the upper bed temperature grid. The resistances
were matched by construction such that the external standard
resistance at room temperature had the same resistance as the
temperature recording grids at 212 degrees Fahrenheit. By doing
this and using the circuit shown in Plate VI, it was possible
to expand any portion of the 100 millivolt range of the instru-
ment to a full scale reading. This feature greatly increased
the accuracy of the instrument over the span of temperatures
used throughout the drying oven.

The lower temperature of each temperature span used was
made to read zero on the millivolt scale by applying a "bucking"
potential on the circuit with a portable potentiometer. From
the potentiometer that supplied the bucking potential the dif-
ference in potential between the temperature recording grids
and the bucking potential was introduced to a General Electric
self-balancing potentiometer. The input to the self-balancing
potentiometer could not exceed 10 millivolts, and to control
this a variable resistance or decade box was placed in that
portion of the circuit that contained the standard resistor
and temperature recording grids.

PLATE VI
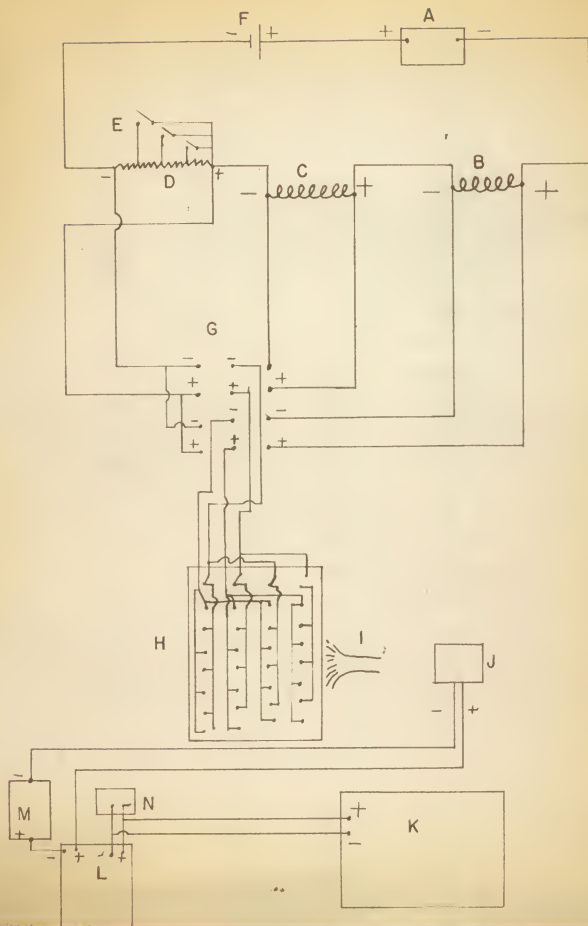
Since the output of the General Electric potentiometer was current (in milliamperes) instead of potential (in millivolts), as required for the operation of the Brown multi-point recorder, and to facilitate the spreading of the range, a variable resistance or decade box was placed across the output terminals of the General Electric instrument.

Ohms law states that $E = IR$, therefore, for any given current, I, an increase in resistance, increased the resulting output voltage, E. Likewise, a decrease in the resistance resulted in a decreased output voltage for the same current applied. Therefore, to spread the scale reading for a given potential difference between the temperature recording grids and the applied bucking potential required only the proper adjustment of the variable resistance.

For each temperature span used, the settings for each of the decade boxes and the portable potentiometer was calculated. The calculations were as follows for the 450 to 212° F. span:

Resistance of bottom grid (G_1) at 212° F. = 1.142 ohms

Resistance of top grid (G_2) at 212° F. = 1.141 ohms

$R_t = R_o (1 + \alpha T)$

For grid 1:

$$\frac{R_{450}}{R_{212}} = \frac{1 + 0.002315 \ (T_{450} - 32)}{1 + 0.002315 \ (T_{212} - 32)}$$

$$R_{450} = 1.142 \ (\frac{1.97}{1.412}) = 1.595 \text{ ohms at } 450° \text{ F.}$$

For grid 2 (top grid):

$$\frac{R_{450}}{R_{212}} = \frac{1 \neq 0.002215 \ (T_{450} - 32)}{1 \neq 0.002215 \ (T_{212} - 32)}$$

$$R_{450} = 1.141 \ (\frac{1.928}{1.394}) = 1.579 \text{ ohms at } 450^\circ \text{ F.}$$

If the setting on the decade box in series with the grids equal to 250 ohms were assumed,

$$I = \frac{E}{R}$$

$$I = \frac{1.5 \text{ (voltage of dry cell)}}{1.595 \neq 1.579 \neq 1.142}$$

$$I = \frac{1.5}{254.316} \text{ or } 0.005895 \text{ amperes.}$$

Since resistance of bottom grid had the higher value at 212° F., the calculation for the portable potentiometer setting was based upon the resistance of the bottom grid.

$$E = IR$$

$$E_{G_1} = (0.005895) \ (1.142) - 6.74 \text{ millivolts or the}$$
setting of the portable potentiometer.

For grid 1:

$R_{450} - R_{212} = 0.453$ ohms - the resistance to provide

100 millivolts deflection on the Brown recorder.

$E = IR$

$E = (0.453) \ (0.005895)$

$E - 0.002668$ volts at 450° F. or 2.668 millivolts

fed to self balancing potentiometer to give 100

millivolt deflection on the Brown recorder.

For 2nd decade box setting:

$$\frac{100}{2.668} = 37.5$$

$37.5 \times 2 = 75$ ohms at 450° F.

MATERIALS

Alfalfa used in this project was grown in the Imperial
Valley of California. This alfalfa was used because this re-
search was carried out during the winter months when fresh
alfalfa was unavailable in this section of the country. The
alfalfa was cut in November, 1952, immediately packaged and
placed in a deep freeze at a temperature of minus 20 degrees
centigrade. After being in the deep freeze for sufficient
time, the alfalfa was removed and packed in dry ice for Air
Express shipment to the Chemical Engineering Department of
Kansas State College. Upon arrival, the frozen material was
placed in a deep freeze locker of the chemistry department un-
til ready for use.

METHODS

A preliminary study indicated that the time interval be-
tween removal from deep freeze storage and dehydration had an
effect on the carotene content retention; therefore, a stand-
ard interval of ten minutes out of storage was used.

Each run was made using 50 grams of alfalfa. This alfalfa
was chopped by hand, and care was taken to separate each in-
dividual particle. The alfalfa was mixed thoroughly to assure
a typical amount of stems and leaves in each sample. The
sample was placed on the static drying bed and lowered into
the drying oven. The alfalfa was then dehydrated in the con-
tinuous drying oven for a desired period of time.

Before placing the alfalfa in the dehydrator, the drying
equipment was heated to operating temperatures and the controls
were adjusted. This preliminary heating reduced the lag in
reaching the maximum drying rate. As soon as the alfalfa was
placed in the dehydrator, the 16-point Brown temperature re-
corder was started. For drying at varying temperature spans,
the power input to the heater was varied from the beginning to
the end of each run. Upon completion of the drying time, the
alfalfa was removed from the dehydrator and the final weight
recorded. The dehydrated material was then ground in a hammer
mill and a final moisture and carotene content analysis made.

Both the initial moisture content of the green alfalfa
and the final moisture content of the dehydrated alfalfa were
determined. This was done by placing samples in a vacuum oven
and dried until no further change in weight was observed.

A simplified method for the determination of carotene in
dehydrated alfalfa was used. This method was presented at the
107th. meeting of the American Chemical Society by Ralph E.
Silker, W. G. Schrenk, and H. H. King of the Chemistry Depart-
ment of Kansas State College (7). Two grams of finely ground
alfalfa was weighed into a glass stoppered Erlenmeyer flask and
60 ml. of a one to two mixture of acetone and Skellysolv-B was
added. The flask was shaken and the contents then set aside
in the dark for 16 to 18 hours. The extract was then filtered
through a Buchner funnel, and the residue washed, by decantation,
with several small portions of Skellysolv-B. The filtrate was
transferred to a beaker and was heated on a steam cone to drive

off most of the remaining acetone. This was concentrated to approximately 40 ml. The concentrate was drawn through a column consisting of two parts, Hyflo Super-cel and one part magnesia (micron brand number 2461). The carotene was then eluted from other pigments with 4 percent acetone-Skellysolv-B solution. The eluate was brought to a volume of 250 ml. with Skellysolv-B. The sample was then read on the Beckman Spectrophotometer.

The method used for the determination of carotene from the fresh green alfalfa was that of Moore and Ely (4). Five grams of fresh alfalfa was weighed into a beaker and was blanched for one minute with steam. To the blanched alfalfa was added 200 ml. of a one to three mixture of Skellysolv-B and ethyl alcohol. The next step consisted of comminuting the alfalfa and mixture in a Waring blendor for three minutes. The extract was filtered through a Buckner funnel and the residue washed by decantation, with small portions of Skellysolv-B. The filtrate was then transferred to a separatory funnel, and the extract was separated. The extract was transferred to a beaker and heated on a steam cone to drive off the ethanol. This was concentrated to approximately 30 ml. The concentration was then drawn through a column consisting of two parts Hyflo Super-cel and one part magnesia (micro brand number 2461). The carotene was eluted from the other pigments with 4 percent solution of acetone and Skellysolv-B, and the eluate brought to a volumn of 250 ml. with Skellysolv-B. The sample was read on the Beckman Spectrophotometer.

DATA

A total of 26 runs were made, 15 of which were with super-
heated steam. The remaining 11 were with air as the drying medium.
The data taken on each run are presented in Tables 1 to 8, and
the plots for data are presented in Figures 2 to 5. In Fig. 2
is shown the effect of drying time on the retention of the caro-
tene content, and in Figs. 3 to 5 is shown the temperature and
time curves for runs 12, 23, and 26. Three series were included.
In one, consisting of run 12-20, the temperature was varied from
450° to 212° F. with superheated steam as the drying medium.
Time was also varied for this series. In another, the tempera-
ture was held constant, and the time was varied, using steam as
the drying medium at 375° F. In the third series, comprising
runs 26-36 the temperature and time was varied, with air at a
temperature span of 260° to 115° F. used as the drying medium.

The rate of steam and air for each run was held constant.
The rate of steam was 80 lbs/hr. and the rate of air was 129.5
lb/hr. The same flow conditions for both air and steam were
used. This was made possible by equating the Reynold's number
for each.

$$\left(\frac{D_p \, G}{\mu}\right)_{steam} = \left(\frac{D_p \, G}{\mu}\right)_{air}$$

Since D_p (equivalent diameter of particle) is the same for
both air and steam,

Table 1. Superheated steam used as the drying medium.

Run no.	Time: min.	Carotene content: mg/gr (green)	Carotene content: mg/gr (dry)	Moisture %: (green)	Moisture %: (dry)	Color of dry alfalfa	% loss of carotene
12	15	0.2160 0.2250	0.2041 0.2080	68.3	8.6	gray	5.50 7.55
12A	16	0.2170 0.2140	0.1625 0.1685	70.1	3.23	light brown	25.00 20.25
13	17	0.2425 0.2845	0.0791 0.0810	69.8	0.0	burnt	68.50 71.40
14	14	0.2150 0.2358	0.2105 0.2158	71.0	11.6	gray green	2.10 8.50
15	13	0.2195 0.2180	0.1870 0.2180	70.5	11.5	gray green	14.80 0.00
16	11	0.1815 0.2300	0.2255 0.2345	70.2	19.6	green	gain gain
17	12	0.2220 0.2325	0.2210 0.2175	68.9	15.15	light green	0.455 6.45
18	15	0.1810 0.2180	0.1735 0.1745	69.9	9.6	gray	4.14 20.00
19	14	0.2165 0.1875	0.1965 0.2090	70.1	10.7	gray green	9.24 gain
20	16	0.2215 0.2040	0.1645 0.1685	69.8	4.73	gray	25.80 17.40

Table 2. Superheated steam used as the drying medium with the temperature constant.

Run no.	Time: min.	Carotene content: mg./gr (green)	Carotene content: mg./gr (dry)	Moisture %: (green)	Moisture %: (dry)	Color :dry alfalfa	:% loss of :carotene
21	15	0.2880	0.1175	69.8	0.0	burnt	59.6
		0.2952	0.1295				56.2
22	11	0.3471	0.1380	71.38	0.55	burnt	60.3
		0.1546	0.1356				12.35
23	9	0.2180	0.1750	69.7	6.25	light brown	19.7
		0.2150	0.1610				25.0
24	9	0.2063	0.1764	71.25	8.75	gray	14.55
		0.2842	0.1782				37.4
25	8	0.2130	0.2123	69.45	14.1	gray	0.0
		0.2334	0.2213				0.515

Table 3. Air used as the drying medium

Run no.:	Time: min.	Carotene content: mg/gr (green)	Carotene content: mg/gr (dry)	Moisture %: (green)	Moisture %: (dry)	Color of: dry alfalfa	% loss of :carotene
26	15	0.2431 0.2123	0.1110 0.1321	70.45	8.2	green	54.0 37.7
27	16	0.3192 0.1415	0.1240 0.1252	68.7	3.1	green	61.0 11.35
28	17	0.2360 0.2460	0.1320 0.1340	70.5	1.5	light green	44.1 45.5
29	18	0.2281 0.2252	0.1172 0.1312	71.3	0.5	light green	48.7 41.7
30	14	0.2070 0.2852	0.1330 0.1342	71.5	12.1	green	35.8 53.0
31	13	0.2590 0.2304	0.1150 0.1384	69.8	11.3	green	55.6 40.0
32	12	0.1918 0.2642	0.1688 0.1632	68.9	15.5	green	12.0 38.3
33	16	0.2681 0.2380	0.1021 0.1030	69.9	4.95	green	62.0 56.8
34	15	0.2011 0.2760	0.1312 0.1360	70.4	10.2	green	34.8 50.5
35	14	0.2040 0.2220	0.1330 0.1330	69.8	11.2	green	34.7 39.6
36	8	0.2180 0.2235	0.1740 0.1415	69.9	15.2	green	20.0 36.5

Table 4. Time and temperature correlation.

Time min.	Run no. 12 T1	T2	Run no. 12A T1	T2	Run no. 13 T1	T2	Run no. 14 T1	T2	Run no. 15 T1	T2
1	445	280	445	295	448	308	444	280	439	294
2	434	285	428	315	442	315	420	298	429	315
3	423	302	400	334	432	324	400	315	412	328
4	399	315	384.5	338	419	325	380	318	378	330
5	375	321	362.5	326	395	325	358	317	357	326
6	332	312	347.4	315	381	315	341	317	323	318
7	310	302	330	300	361.5	304	332	315	310	306
8	297	290	310	288	340	300	318.2	302	293	291
9	290	271	295	272	327	290	298	290.5	273	270.5
10	276	260	278	260	311	280	281.5	277	260	258
11	263	250	263	245	295	262.5	267	262.5	245	244
12	252	238	243	232	275	254	243	242	234.5	233.5
13	260	228	232.5	225	260	243	228.5	228	218	218
14	238	221	225	217	248	232	216.5			
15	228.5	215	217	215	233	224				
16	221		215		224	215				
17	215				215					

Table 5. Time and temperature correlation.

Time min.	Run no. 15 T1	T2	Run no. 17 T1	T2	Run no. 18 T1	T2	Run no. 19 T1	T2	Run no. 20 T1	T2
1	447	276.5	445	284.5	445	286	443	281	444	296
2	442	300	425	311	422	300	421	299	429	314
3	372	309	402.5	319.5	400	311.5	400	315	400	335
4	351	308	375	323.5	374	320	381	317	385	338
5	325.5	298.5	346	322.5	333	312	359	316	363	337
6	303	285.5	319	304	311	301	340	315	348	336
7	281.5	272.5	299	291	298	290	330	312	330.5	316
8	262.5	259	278	270	275.5	272	300	300	309	300
9	247	246	253	251	263.5	260	317.5	290	294.5	286
10	236	235	240	239	252	250	282	278	279	274
11	222	221	224.5	224	238.5	237.5	266	261	264	261
12			217	217	228.5	228	242	241	248	246
13					223	222	229.5	229	232.5	231
14					219	219	217	217	227	226
15					214	214			218	218
16									216	216

Table 6. Time and temperature correlation.

Time: min.	Run no. 21		Run no. 22		Run no. 23		Run no. 24		Run no. 25	
	T_1	T_2	T_1	T_2	T_1	T_2	T_1	T_2	T_1	T_2
1	375	254	375	254	375	254	375	247.3	375	251
2	375	280.5	375	268.5	375	286.5	375	287.8	375	286.5
3	375	309.5	375	311.5	375	308.5	375	304.5	375	307.5
4	375	339	375	330	375	329	375	330	375	330
5	375	348	375	352	375	343.5	375	347	375	346.5
6	375	359.8	375	360	375	358	375	360	375	359.5
7	375	368	375	366	375	364.5	375	366.5	375	365.5
8	375	374	375	371	375	370	375	371	375	371
9	375	375	375	375	375	375	375	375		
10	375	375	375	375			375	375		
11	375	375	375	375			375			
12	375	375	375	375						
13	375	375	375	375						
14	375	375	375	375						
15	375	375	375	375						

Table 7. Time and temperature correlation.

Time min.	Run no. 25		Run no. 27		Run no. 28		Run no. 29		Run no. 30	
	T_1	T_2	T_1	T_2	T_1	T_2	T_1	T_2	T_1	T_2
1	260	151	262	138	262	129	262	142.5	262	124.5
2	258	172	260	173.5	260	166	262	185	258	161
3	24?.5	186	258	191	258	190	260	211.5	255	177.5
4	234.5	190	248	199	254	199.5	258	230	246	188.5
5	215	186	236.5	201	239	200	255	234	231	190
6	198	174.5	227	200	224	194	249	228	217	186.5
7	173.5	160	217.5	196	208.5	189	228	216.5	201.5	181
8	155	144	205	190.5	197	184	212	203	179	166.5
9	141	132.5	184.5	174.5	185	173.5	191	185	157	147.5
10	134	125	163	155	175	161.5	173	166.5	139.5	130
11	126.5	122.5	144.5	136	159	150	153	150	131.5	125
12	123.5	120.5	133.5	129.5	138.5	133	136	133.5	125.5	121
13	123.5	119	127.5	125	128.5	125	129	126	121	119.5
14	118.5	117.5	123.5	120	125.5	123	124	123.5	117	116
15	116.5	115.5	120	118	123	121	121	120.5		
16			117.5	117	121	120	119	119		
17					119	118	118	118		
18							116.5	116.5		

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Table 8. Time and temperature correlation.

Time min.	Run no. 31 T_1	T_2	Run no. 32 T_1	T_2	Run no. 33 T_1	T_2	Run no. 34 T_1	T_2	Run no. 35 T_1	T_2	Run no. 36 T_1	T_2
1	258	133.5	260	136	262	137	262	148	262	130	260	132
2	255	170	257	170	259	172	255	171.5	258	162	251	160
3	250	201	250	187	256.7	190	248	187.5	255	180.5	215	177
4	241	205	228	187	246	198.5	233	189.5	247	190.5	172	170
5	225	197.5	201.5	176.5	234.5	199.5	213	185	233	193	155	148
6	205	181	180.5	161.5	223	199.5	196	175.5	220	187.5	133	129.5
7	188.5	166.5	156	146.5	216	197.5	175	161	204.5	180	122.5	120.5
8	161	146	139	131	201.5	188.5	156.5	145	180	167	118	117.5
9	142	130	132	126	180.5	172.5	143	133.5	156.5	148		
10	130	126	126.5	122	159	153.5	135	126	138	131.5		
11	124	121	122	119	137	135	127.5	123	130	125.5		
12	119	117	118.5	117	131	128.5	124	121	124	122		
13	116	115			126	124.5	122	119.5	121	119.5		
14					122.5	121.5	119	118	117	116.5		
15					119.5	118	118.5	117				
16					117	117						

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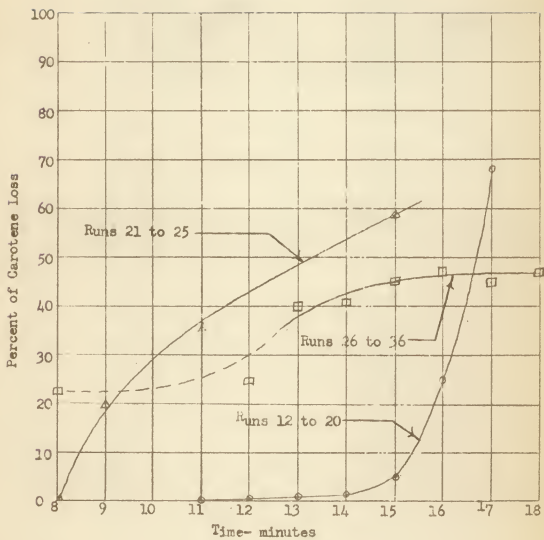
Fig. 2 - Percentage of Carotene Loss Due to Drying
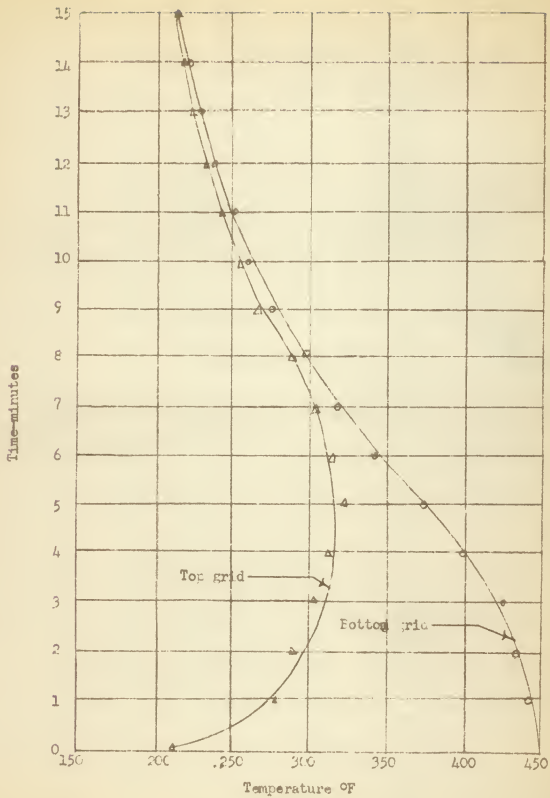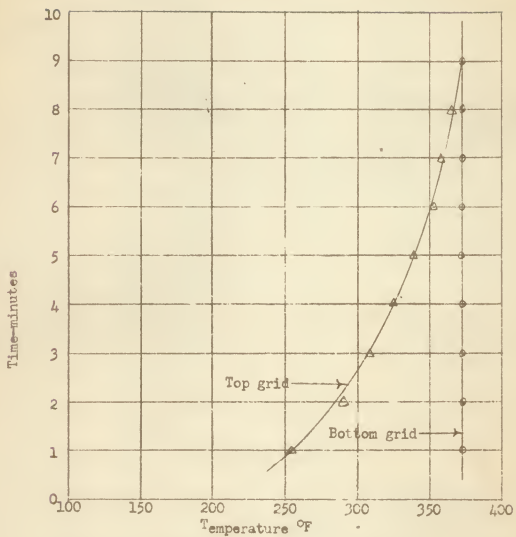(Average values from Tables 1-111)

Fig. 3 — Time versus Temperature Plot for Run 12
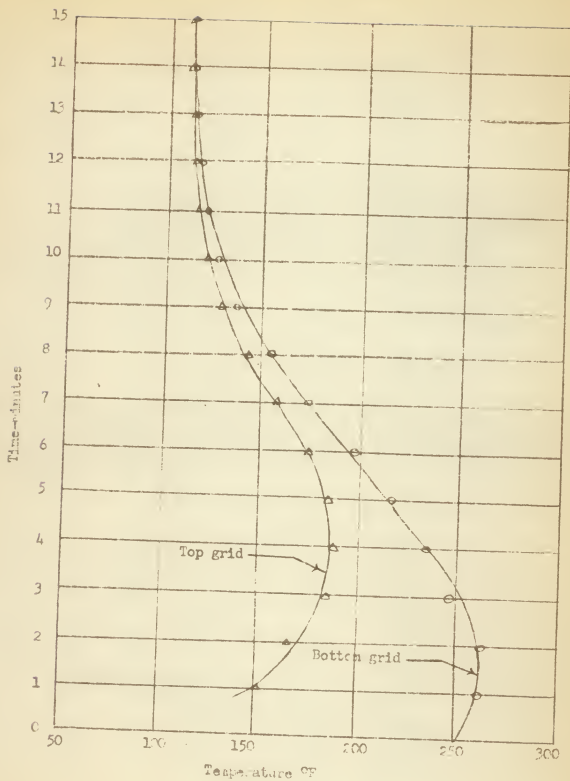
Fig. 4 – Time versus temperature for Run 23

Fig. 5 – Time versus Temperature for Run 26

$$\frac{(G)}{(\mu)}\text{steam} = \frac{(G)}{(\mu)}\text{air}$$

$$G_{\text{air}} = \frac{(G)}{(\mu)}\text{steam}\,\mu_{\text{air}}$$

$$G_{\text{air}} = \frac{80}{0.0175}\ (0.0283) = 129.5\ \text{lb/l hr.}$$

The temperature span for air was determined experimentally. The lower limit of the temperature span was assumed to be 115° F. Since the final moisture content of the alfalfa at the end of 15 minutes for steam drying was known, trial runs using air at a flow rate of 129.5 lb/hr. were made until the desired temperature span was established. A value of 260° F., for the upper limit of the temperature span was determined to be the desired temperature for the air at the initial conditions.

By varying the temperature during a run, a continuous flow process of alfalfa was, in effect, established. This increased the drying time but gave conditions applicable to the present industrial commercial drying processes. The constant temperature rate of drying established a batch process, and drying times were much less than for the varying temperature conditions;(however, higher surface temperatures of the alfalfa will occur).

Runs 1 to 11 were discarded because of inconsistencies arising from measurements of temperature, moisture content, and carotene content. Since the carotene content was the primary important factor, any error in its evaluation rendered the data useless.

Drying rate curves were not used, except as reference, because of the difficulties encountered in the energy balance around the drying section of the oven. The information to be gained from drying rate curves was less advantageous than that

gained by using superheated steam in view of the purpose of the
experimental investigation.

CORRELATION OF DATA

The values of carotene content in mg/gr. as shown in Tables
1 to 3 were calculated from the readings of the Beckman spectro-
photometer. The meter reading of the spectrophotometer were in
mg/liter. Since the volume used in each determination was 250 ml.,
each meter reading for each sample determined was divided by four.
This gave the carotene content in milligrams per liter volume.
The desired values were for milligrams per gram. By the moisture
determination, the number of actual grams of alfalfa for each
sample was determined, and by dividing the value calculated in
milligrams per liter by the number of grams of alfalfa calculated
by knowing the moisture content, the value of carotene content
in mg/gr. was determined.

Sample calculation (using Run 12, dry):

Meter reading $= 1.520$ mg/L

$\frac{1.520}{4} = 0.380$ mg/L

Moisture content $= 8.60$

Weight of alfalfa used $= 2$ grams

$2 \times 0.086 = 0.132$ grams of H_2O in sample used

$2 - 0.132 = 1.868$ grams of alfalfa in sample

0.380 mg/L $\div 1.868$ grams $= 0.2041$ mg/gram

The values of T_1 and T_2 in Tables 4 to 8, were calculated
from the recordings of the 16-point Brown electronic recorder.

Since the recordings were in millivolts, each millivolt represented a fixed value of temperature increase or decrease. This value changed when new settings on the auxiliary equipment of the Brown recorder were made. For runs 12 through 25 the value remained constant, but for runs 26 through 35, new settings were made on the auxiliary equipment, and a new value for these runs determined. In both instances, this fixed value was different for each temperature recording grid.

Calculations for the fixed value of each grid for runs 26 through 35.

At 98 millivolts reading bottom grid temperature = 260° F.

At 81 millivolts reading top grid temperature = 260° F.

At 10 millivolts reading bottom grid temperature = 115° F.

At 2 millivolts reading top grid temperature = 115° F.

98-10 = 88 millivolts = 145° F. for bottom grid

$\frac{145}{88}$ = 1.635° F. for 1 millivolt reading of bottom grid

81-2 = 79 millivolts - 145° F. for top grid

$\frac{145}{79}$ = 1.835° F. for 1 millivolt reading of top grid

By knowing the fixed values for each grid, the determination of T_1 and T_2 values was found by multiplying the value of the millivolt reading by the temperature value for the corresponding grid. The difference in the value for each grid was due to the difference in the temperature coefficient of resistance of the silver wire used in each grid.

SUMMARY OF RESULTS

In Fig. 1, the effect of dehydrating alfalfa with super-heated steam was noted. The percent of loss by using superheated steam was much lower than those when air was used. Only in instances where the dried product was charred or burned with steam, were the percentage of loss greater with superheated steam. This was believed to have resulted from the fact that the surface temperature of the alfalfa using steam was sufficiently high to cause a decomposition of the alfalfa; whereas, for air this was not true. A final moisture content between 12 and 19 percent gave the best results in all instances. The gain in carotene contene of runs 16 and 19 were attributed to more evenly distributed leaf to stem ratio of the ground dry product. Since the leaves contain the greater amount of carotene, a true represented green sample was hard to obtain.

The leaves of the alfalfa contained less moisture content than the stems. Upon drying, the leaves were dried faster and more completely than were the stems. This condition led to a slight burned effect on the leaves below an over-all moisture content of 12 percent and, therefore, the final carotene content of alfalfa below this moisture content was decreased.

Carotene retention of the dry product between 12 and 19 percent moisture for steam was 100 percent in most instances, while for air a loss of 12 to 53 percent resulted.

The color of the final product was quite different for air and steam. A gray or light brown color predominated when steam was used as compared to a natural green color for air. Since a better product was obtained from steam dehydration, the natural green color found for an air-dried product can be sacrificed.

Frozen alfalfa used in this research appeared to exhibit the same characteristics of carotene retention as fresh cut alfalfa.

In addition to offering better carotene retention, steam can offer theoretically more economical dehydration operation because of improved higher thermal efficiences are more possible. Further research on this idea could prove to be useful to the alfalfa dehydration industry.

The surface temperature of the alfalfa in steam dehydration is initially much greater than that of air dried alfalfa. The nutritional value of the two products are indicated to be different by studies being carried out by the Poultry Department of Kansas State College. Studies are still in progress, but alfalfa reaching the higher surface temperature during dehydration appears to offer the best nutritional value. Further experimental studies are required to prove the value of steam as compared to air in this respect.

No further study is believed to be warranted to determine the effect of dehydrating alfalfa with superheated steam on the carotene content as compared with air.

ACKNOWLEDGMENTS

BIBLIOGRAPHY

(1) Griffith, R. B., and C. R. Thompson, "Factors Affecting Destruction of Carotene in Alfalfa," Alfalfa Abstracts, 1950.

(2) Hart, E. B., and R. C. Mills. Jour. Dairy Science, vol. 28, p. 239, 1945.

(3) Mitchell, H. L., and S. M. Houge. Jour. Biol. Chem., vol. 163, pp. 7-14, 1946.

(4) Moore, L. A. and R. Ely. Ind. Eng. Chem., Anal. Encl., vol. 13, p. 600, 1941.

(5) Nelson, W. L., J. K. Loosli, Glen Lobgreen, and Naomi Yoger. "Carotene Retention in Alfalfa Meal," Ind. Eng. Chem., vol. 40, pp. 2196-8, 1948.

(6) "Nichrome and other High Nickel Electrical Alloys," catalog R-49, Driver-Harris Co., Harriston, N. J.,1949.

(7) Silker, R. E., W. G. Schrenk, and H. H. King. Ind. Eng. Chem., Anol. Ed., vol. 16, p. 513, 1944.

(8) U.S.D.A., 1950 Report of Chief of Bureau of Agriculture and Industrial Chemistry, p. 53, 1950.

(9) Wenzel, Leonard, and Robert R. White, "Drying Granular Solids in Superheated Steam," Ind. Eng. Chem., vol. 43, pp. 1829-37, 1951.

APPENDIX

NOMENCLATURE

A   numerical factor

C   orifice coefficient

C_{p_m} molal mean heat capacity

D_p diameter in inches

E   amps

$^\circ F$ degrees Fahrenheit

G   flow rate lb/hr.

gr   grams

H   square root of maximum pressure differential in inches of mercury under water

I   volts

K   density factor

K_1 quality of steam

KW   Kilowatts

l   liters

mg   milligrams

Q   heat required in B.T.U.

R   resistance in ohms

R_t resistivity at any temperature

R_o resistivity at 32° F.

T   temperature $^\circ F$

T_1 temperature $^\circ F$ of bottom temperature recording grid

T_2 temperature $^\circ F$ of top temperature recording grid

t   time in minutes

α temperature coefficient of resistance

μ viscosity in centipois - gr./cm. sec.

DEHYDRATION OF ALFALFA WITH SUPERHEATED STEAM

by

WENDELL J. BEANE

B. S., Indiana Technical College, 1952

AN ABSTRACT

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requirements for the degree

MASTER OF SCIENCE

Department of Chemical Engineering

KANSAS STATE COLLEGE
OF AGRICULTURE AND APPLIED SCIENCE

1953

The purpose of this research was to compare the effect of dehydrating alfalfa with superheated steam on the carotene contene with the effect produced by present drying mediums used for dehydration. Air, under identical flow conditions as that used for steam, was used for the comparative studies.

The ability to perform the necessary dehydration was made possible by design and construction of a dehydrator. An electric heater, capable of producing 950 degrees Fahrenheit superheated steam, was used to supply heat to the drying mediums. Voltage to the heater was regulated by a powerstat, thus allowing the heat supplied to be controlled to the desired amount. Temperature recordings of the drying mediums entering and leaving the drying section were made using a 16-point Brown Electronic Recorder.

Initial and final carotene content analysis was made using a Beckman Spectrophotometer. Superheated steam dehydration showed an increase of plus or minus 30 percent carotene retention as compared to air dehydration. A gray or light brown color predominated when steam was used as compared with air.

Further studies are recommended for steam dehydration. In addition to offering better carotene retention, steam can offer theoretically a more economical dehydration operation because of improved higher thermal efficiencies are more possible. Further research on this idea could prove to be useful to the alfalfa dehydration industry.

The results of these studies should contribute to a better method for carotene retention in dehydrated alfalfa.